CHAPTER 5

RESULTS, DATA ANALYSIS AND DISCUSSION

The results of sintering of tin base bearing alloy (with composition 70% tin, 20% copper and 10% antimony) are measured in terms of its density, porosity and hardness. The physical appearance and microstructure are also analyzed.

5.1 Physical Appearance

Generally, all the specimens remain the same before and after sintering. However, there are a few exceptions according to the type of sintering

a) Microwave sintering

The colors of the specimens that are microwave sintered in air remain the same before and after sintering. This may be because of the fact that the heat is produced inside the specimens at localized spots due to microwaves penetration and then the heat transfers to the outside surface. Therefore there are less chances of oxidation of the specimens. While in conventional heating, the heat from the furnace chamber transfers to the specimens through radiation, the outside surface heats up first and then heat is transferred to the inside of the specimens. In this process the outside heated surface remains in contact with the furnace air for longer period. Therefore, there are greater chances of oxidation of the specimens in conventional sintering process, if not performed in a neutral atmosphere. Some

indentation is observed at the middle of the specimens. This might be because of the thermocouple touching the specimen and thus leaving a burn mark. Otherwise, the surface finish is quite smooth.

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b) Conventional sintering

The colors of the specimens that are conventionally sintered in Argon gas environment remain the same before and after sintering with the exception of one specimen. The specimen compacted at 156MPa pressure and sintered at 220°C for 60 minutes turned into grey color. Argon gas is used during the sintering process and inert gasses are supposed to be non-reactive. The reason for this compact turning gray may be that at low compaction pressure, air might already be there

inside the pores. When sintered at high temperature for a prolonged period of time, the specimen seems to have oxidized even in argon gas atmosphere in the furnace.

5.2 Dimensional Changes

The dimensional changes of the specimens before and after sintering are observed. However, only the diameter of the specimens is put into consideration because a sintering compact can shrink. The height or the thickness of the specimens is not considered. Figure 5.2 shows the average values of before and after sintering while Table 5.2 shows the percentages of the diameter change. Figure 5.2.1 shows the average values of percentage diameter change after sintering.

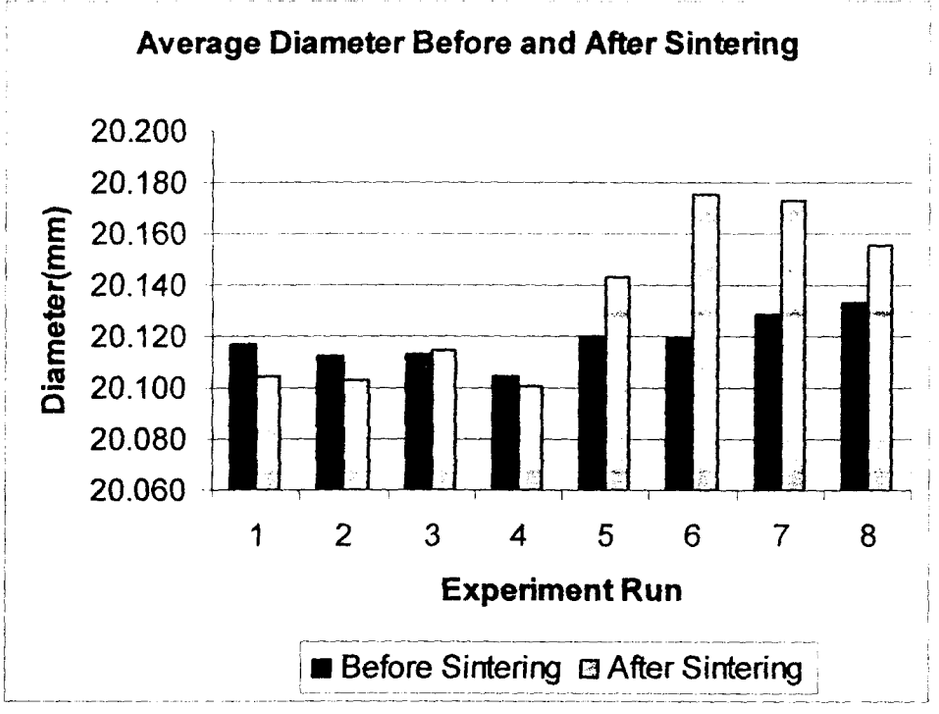

Figure 5.2 Bar chart showing the average diameter values of before and after sintering

Experiment Run	Experiment Condition	A (°C)	B (MPa)	C	D (min)	%of diameter change			
						y_1	y_2	y_3	Avera
1	$A_0B_0C_0D_0$	140	156	microwave	30	-0.229	0.000	0.050	-0.06
2	$A_0B_0C_1D_1$	140	156	Ar gas	60	0.119	-0.119	-0.134	-0.04
3	$A_0B_1C_0D_1$	140	312	microwave	60	0.070	0.015	-0.065	0.01
4	$A_0B_1C_1D_0$	140	312	Ar gas	30	0.080	0.035	-0.164	-0.02
5	$A_1B_0C_0D_0$	220	156	microwave	30	0.184	0.065	0.099	0.12
6	$A_1B_0C_1D_1$	220	156	Ar gas	60	0.329	0.298	0.219	0.28
7	$A_1B_1C_0D_1$	220	312	microwave	60	0.398	0.035	0.234	0.22
8	$A_1B_1C_1D_0$	220	312	Ar gas	30	0.249	0.000	0.084	0.11

Table 5.2 Percentage of diameter change

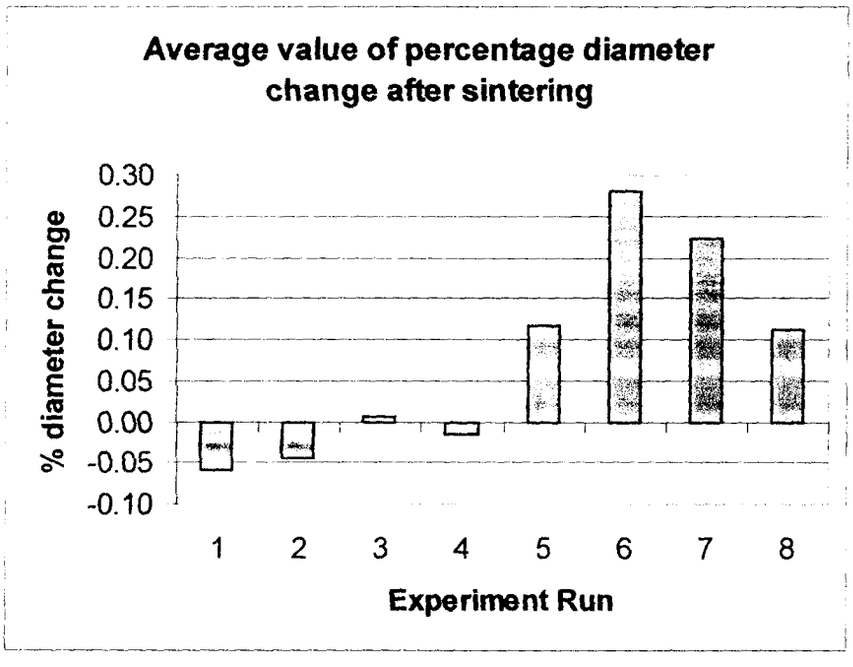

Figure 5.2.1 Bar chart showing mean values of percentage diameter change after sintering

From Figure 5.2 it can be observed that the diameters of the green compacts are larger than the die diameter in which the specimens were compacted. This is because of the

elastic recovery of some of the powder particles, which were in the plastic range. In general, the elastic recovery is more for the specimens compacted at higher pressures (312 MPa) as compared to the specimens compacted at low pressure (156 MPa).

From Figure 5.2.1 it is observed that when sintered at high temperature (220°C), expansion occurs irrespective of type of sintering (Experiment no. 5, 6, 7 & 8), while sintering at low temperature (140°C) results in shrinkage of the specimens. Microwave sintering of specimens compacted at 312 MPa, at 140°C for 60 minutes (Experiment no. 3), gives best dimensional accuracy. Conventional sintering of specimens compacted to the same pressure in argon gas atmosphere at 220°C for 60 minutes gives the worst dimensional accuracy (Experiment no 6). Under the condition of Experiment 3, the density ratio is highest (0.95, Graph 5.3.2), open pores porosity is the least (0.02, Table 5.4). But under this condition hardness of the specimen is low (22.2 VHN, Table 5.5). This may be because the hard copper-antimony inter-metallic phase does not seem to be fully formed and not as uniformly distributed as in case of conventional heating as is evident from micro structures given in Figure 5.6.2 and Figure 5.6.5 respectively.

Thus, it can be concluded that for the best dimensional accuracy of the products the green compacts shall be sintered by microwave heating and higher compaction pressure, low sintering temperature and longer sintering time shall be used.

It is also observed that under the conditions of Experiment no. 6 largest expansion has occurred. The percentage increase in diameter is 0.28% (Table 5.2). But the density is quite high (7.0 g/cm^3, Table 5.3), even though open pores porosity is 1.8% (Table 5.4), the hardness is the highest (32.9 VHN, Table 5.5). The microstructure of the specimen under this condition is shown in Figure 5.6.5. From the microstructure one can see that there is more purple area in this structure as compared to other

microstructures. This shows that a larger amount of hard inter-metallic Cu-Sb compound phase has formed under this condition. High open porosity may be attributed to a larger pore appearing on the surface. This also shows that the inter-metallic compound may be having larger volume that the constituents, that lead to more expansion of these specimens. Thus, the specimens compacted at lower temperature can give higher hardness if they are conventionally sintered at higher temperature for a longer time period in argon gas atmosphere.

5.3 Density

Table 5.3 shows the density of each experimental run and the SN ratio. Since the objective is to get higher density, the larger-the-better characteristic is used to calculate the SN ratio. The way to compute SN ratio is:

$$SN_i = -10\log\left(1/n\sum_{j=1}^{n} 1/y_{ij}^2\right) \qquad \text{----------Equation (2)}$$

where n = number of experiment

$\sum y_{ij}$= density value in each row (i^{th} experimental conditions of control factors)

Sample calculation of SN ratio for the first experimental run is given below:

By referring to Table 5.3, the values are:

n = 3; $y_1 = 7.096$; $y_2 = 7.119$ and $y_3 = 7.006$

Therefore, $SN_1 = -10\log [(1/3)\ (1 / (7.096)^2 + 1/ (7.119)^2 + 1/ (7.006)^2 = 16.992$

Array type	Inner array (L_8)							Observations					
	Control factor assignment and *column number*							Raw data Density (g/cm^3)					SN(based on larger-the-better character-istic)
	A	B	AxB	C	AxC	D	AxD	y_1	y_2	y_3	Average density	Standard Deviation	
Experiment number	1	2	3	4	5	6	7						
1	0	0	0	0	0	0	0	7.096	7.119	7.006	7.074	0.060	16.992
2	0	0	0	1	1	1	1	7.159	5.644	6.730	6.511	0.781	16.139
3	0	1	1	0	0	1	1	7.207	7.058	7.270	7.178	0.109	17.118
4	0	1	1	1	1	0	0	7.285	6.702	7.003	6.997	0.292	16.883
5	1	0	1	0	1	0	1	6.846	7.087	7.094	7.009	0.141	16.910
6	1	0	1	1	0	1	0	7.123	7.083	7.074	7.093	0.026	17.017
7	1	1	0	0	1	1	0	7.070	6.966	7.177	7.071	0.106	16.988
8	1	1	0	1	0	0	1	7.205	7.219	6.729	7.051	0.279	16.951
	Current condition												

Table 5.3 Density of each experimental run and its SN ratio.

5.3.1 The effect of input parameters on density

Figure 5.3.1 to Figure 5.3.1.3 shows the effect of temperature, compaction pressure, type of sintering and sintering time on density, respectively. From the graphs, it can be concluded that density increases as the sintering temperature and compaction pressure increases (Figure 5.3.1 and Figure 5.3.1.1). Microwave sintering yields higher density than conventional sintering (Figure 5.3.1.2). Density decreases as the sintering time increases (Figure 5.3.1.3).

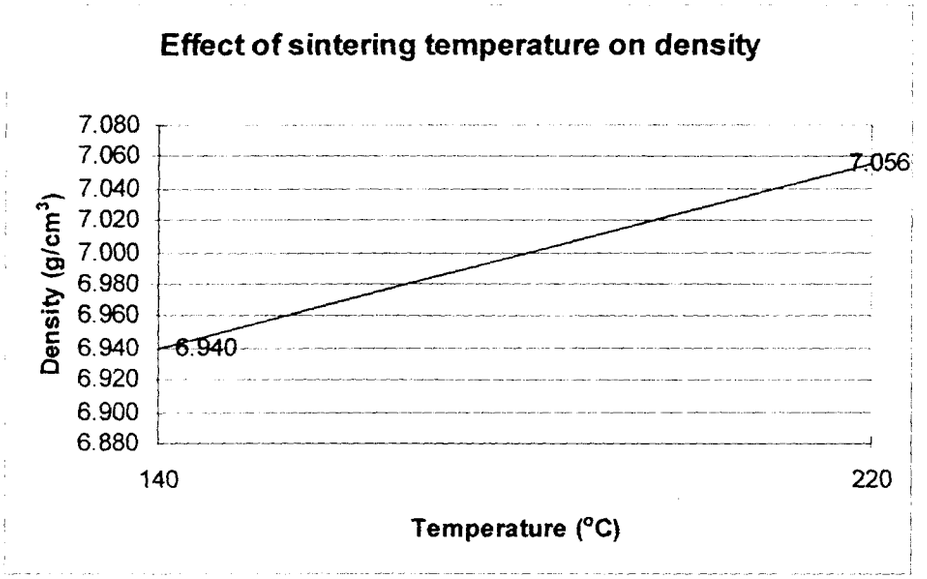

Figure 5.3.1 Effect of sintering temperature on density

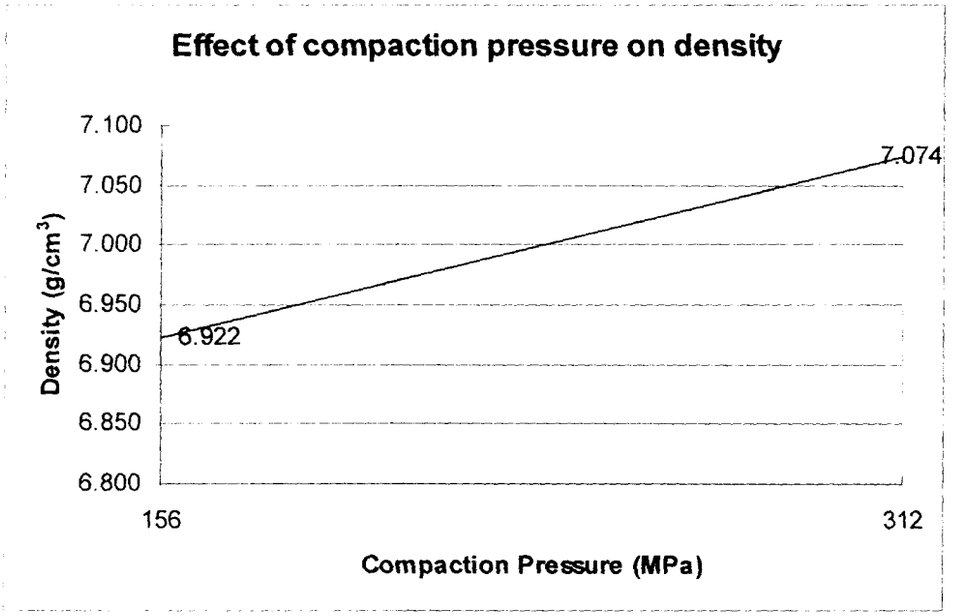

Figure 5.3.1.1 Effect of compaction pressure on density

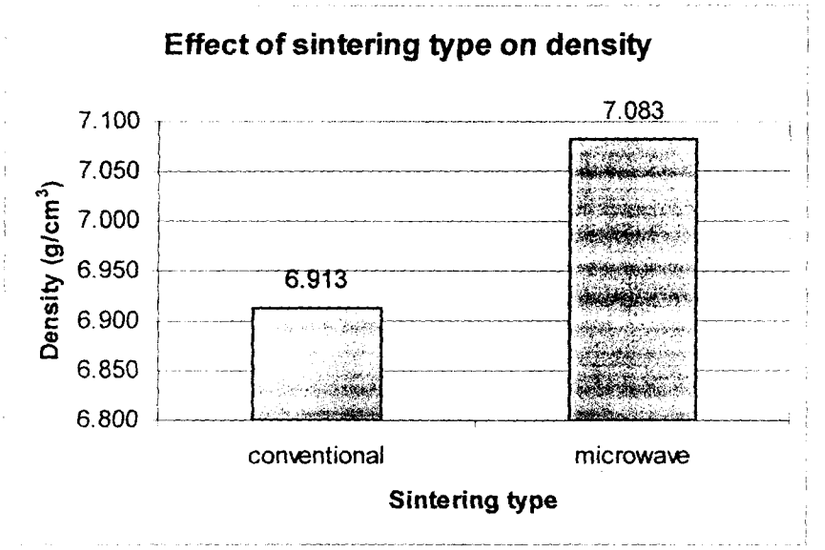

Figure 5.3.1.2 Effect of type of sintering on density

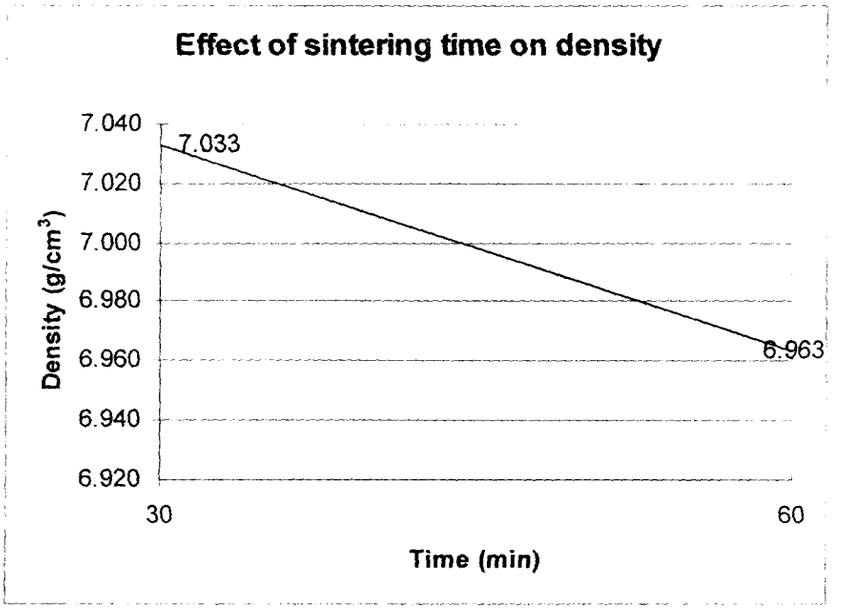

Figure 5.3.1.3 Effect of sintering time on density

5.3.2 Density ratio

The density ratio is shown in Table 5.3.2 and its graph is shown in Figure 5.3.2. The density ratio is calculated as:

$$\text{Density Ratio} = \frac{\text{Actual density}}{\text{Theoretical density}} \quad \text{----------Equation (3)}$$

Where

Theoretical density = 0.7ρ (tin) +0.2ρ (copper) + 0.1ρ (Antimony)

= (0.7*7.29) + (0.2*8.93) + (0.1*6.69)

= 5.1009 + 1.7866 + 0.669

= 7.558g/cm³

Experiment Run	Experiment condition	A	B	C	D	Average actual density (g/cm³)	Density ratio
1	$A_0B_0C_0D_0$	140°C	156MPa	microwave	30 min	7.074	0.936
2	$A_0B_0C_1D_1$	140°C	156MPa	Ar gas	60 min	6.511	0.862
3	$A_0B_1C_0D_1$	140°C	312MPa	microwave	60 min	7.178	0.950
4	$A_0B_1C_1D_0$	140°C	312MPa	Ar gas	30 min	6.997	0.926
5	$A_1B_0C_0D_0$	220°C	156MPa	microwave	30 min	7.009	0.928
6	$A_1B_0C_1D_1$	220°C	156MPa	Ar gas	60 min	7.093	0.939
7	$A_1B_1C_0D_1$	220°C	312MPa	microwave	60 min	7.071	0.936
8	$A_1B_1C_1D_0$	220°C	312MPa	Ar gas	30 min	7.051	0.933

Table 5.3.2 Density ratio

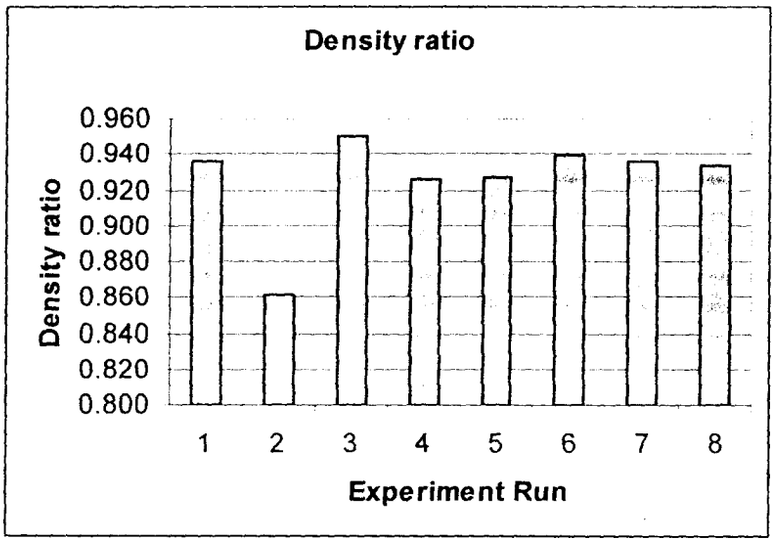

Figure 5.3.2 Density ratio of experimental run

5.3.3 Densification parameter

The densification parameter is shown in Table 5.3.3. The densification parameter is calculated by using the formula below:

$\Psi = (\rho_s - \rho_g) / (\rho_t - \rho_g)$ ---------Equation (4)

Where Ψ = densification parameter

ρ_s = sintered density

ρ_g = green compact density

ρ_t = theoretical density

Experiment condition	A	B	C	D	Green compact density (g/cm^3)	Sintered density (g/cm^3)	Densification parameter
$A_0B_0C_0D_0$	140°C	156MPa	microwave	30 min	6.863	7.074	0.304
$A_0B_0C_1D_1$	140°C	156MPa	Ar gas	60 min	6.844	6.511	-0.466
$A_0B_1C_0D_1$	140°C	312MPa	microwave	60 min	6.923	7.178	0.402
$A_0B_1C_1D_0$	140°C	312MPa	Ar gas	30 min	6.944	6.997	0.086
$A_1B_0C_0D_0$	220°C	156MPa	microwave	30 min	6.87	7.009	0.202
$A_1B_0C_1D_1$	220°C	156MPa	Ar gas	60 min	6.855	7.093	0.339
$A_1B_1C_0D_1$	220°C	312MPa	microwave	60 min	6.912	7.071	0.246
$A_1B_1C_1D_0$	220°C	312MPa	Ar gas	30 min	6.958	7.051	0.155

Table 5.3.3 Densification parameter.

5.3.4 Pareto Anova

The optimum level for each factor will be at which the average of the "iso-level" value of S/N ratio is maximum. The average iso-level value is determined from the values of S/N ratios at low and high levels of the factors. For example, the average S/N ratio for density at low level of factor A is determined from experimental runs 1,2,3 and 4 and for high level from experimental runs 5,6,7 and 8. Similarly, for other factors these iso-levels values are determined and recorded in Table 5.3.4. Figure 5.3.4 shows the iso-level SN values for density at different level of parameters.

Factors	Density at Low Level (0)	Density at High Level (1)	Difference
A Sintering temperature	16.783	16.967	0.18
B Compaction pressure	16.765	16.985	0.22
Interaction AxB	16.768	16.982	0.21
C Type of sintering	17.002	16.748	0.25
Interaction AxC	17.020	16.730	0.29
D Sintering time	16.934	16.816	0.12
Interaction AxD	16.970	16.780	0.19

Table 5.3.4 Average iso-level SN ratio values for density

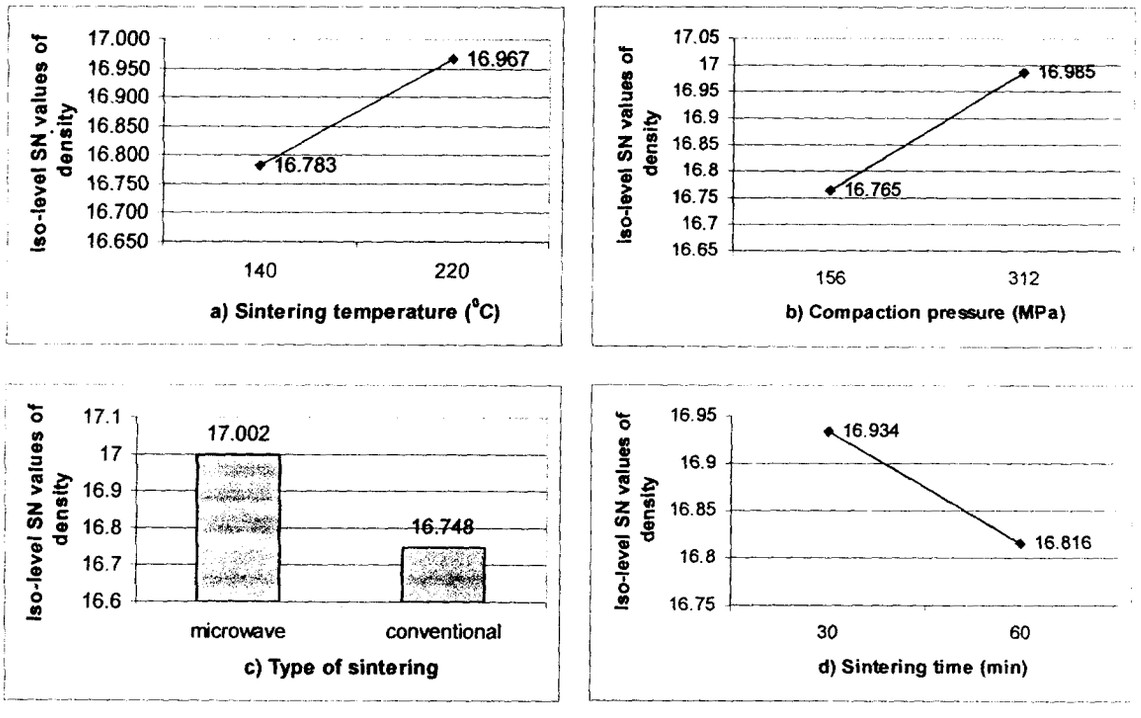

Figure 5.3.4 Iso-level SN values of density at different levels of parameter a)sintering temperature; b)compaction pressure; c)type of sintering; d)sintering time

From Table 5.3.4, one can see that the optimum combination of input parameters for highest density is $A_1B_1C_0D_0$ which is at temperature of 220°C, compaction pressure of 312MPa, conventional sintering and sintering time of 30 minutes. The Pareto

Analysis of Variance (ANOVA) applied to this data gives the same optimum level of controllable factors as the iso-level technique and shown in Table 5.3.4.1.

Table 5.3.4.1 Pareto Analysis of Variance (ANOVA) for density

Factor and interactions		A	B	AxB	C	AxC	D	AxD	Total
Sum at factor level	0	67.132	67.058	67.070	68.008	68.078	67.736	67.880	sum of 0,1 level = 134.998
	1	67.866	67.940	67.928	66.990	66.920	67.262	67.118	
Difference at 2 levels		0.734	0.882	0.858	1.018	1.158	0.474	0.762	
Square of difference		0.5388	0.7779	0.7362	1.0363	1.3410	0.2247	0.5806	5.2355
Contribution ratio (%)		10.29	14.86	14.06	19.79	25.61	4.29	11.09	100.00

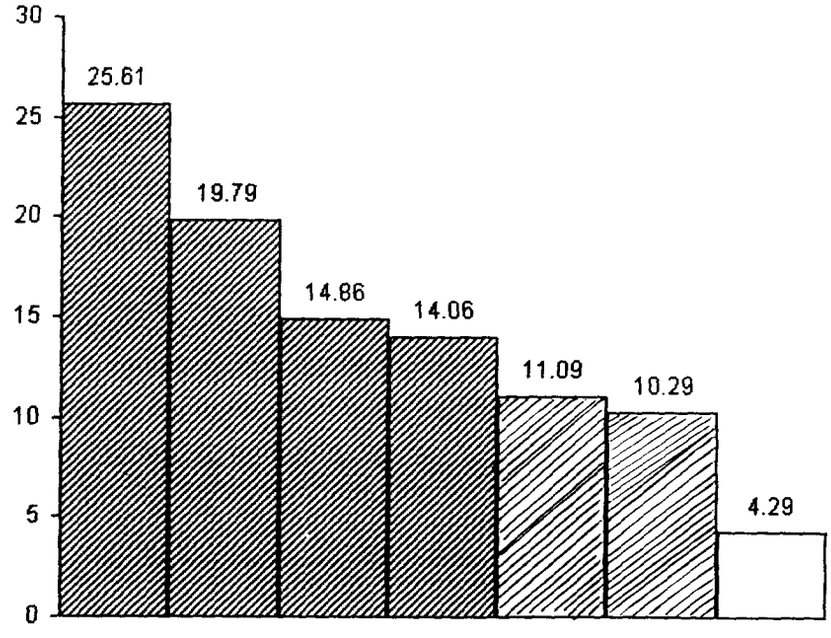

Figure 5.3.4.1 Pareto diagram for density

AxC	C	B	AxB	AxD	A	D	
25.61	45.40	60.26	74.32	85.41	95.70	100.00	

Figure 5.6.7 shows the specimen conventionally sintered at 220°C for 30 minutes and compacted at 312 MPa. The specimen under this condition has swelled with a diameter change of 0.11%. It has 7.051 g/cm^3 density and porosity of 0.18%. Its hardness is 26.72. The grain size of this phase is also larger in this case. The smaller pores have segregated into a large pore with smoother edge. Where as when higher sintering temperature is accompanied with higher compaction pressure and lower sintering time, the hard phase grains are refined to finer sizes and distributed uniformly throughout the specimen, when conventional heating is used as shown in Figure 5.6.7.

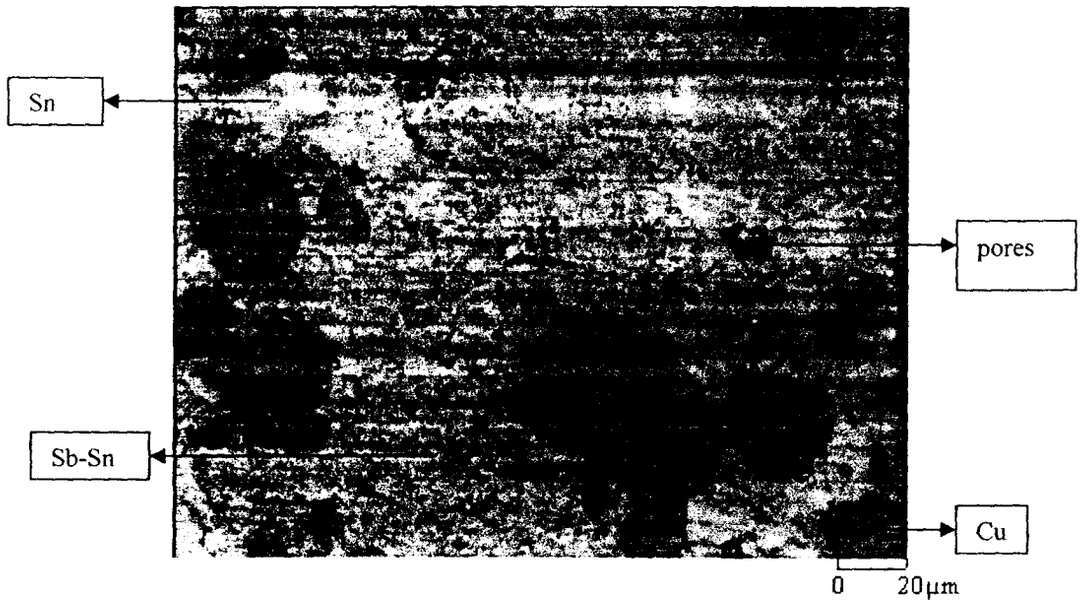

Figure 5.6.7 Microstructure of Experiment 8 (220°C, 312 MPa, Ar gas, 30 min)

Thus it can be concluded that under microwave sintering the hard inter-metallic compounds of tin-antimony and tin-copper do not fully precipitate out. Conventional sintering, high sintering temperature and larger sintering time favor the formation of inter-metallic compound crystals between copper and tin.